Adaption of luting cement to enamel, dentin and restorative material

GUDBRAND ØILO

NIOM, Scandinavian Institute of Dental Materials Oslo, Norway, and Department of Prosthetics, Dental Faculty, University of Oslo, Oslo, Norway

Øilo, G. Adaption of luting cement to enamel dentin and restorative material. *Acta Odontol. Scand.* 36, 149–156

Thirtytwo teeth with artificial crowns cemented with four different cements were sectioned and the cementing interfaces were studied by means of a replica technique. Scanning electronic microscopic examination of the replicas demonstrated that slits occurred in all specimens either at the cement/tooth interface, the cement/alloy interface or both. Differences between the various cements in width and length of the slits could not be assessed by this method, but marked variations in the localization of the slits were noted. A composite resin cement showed slits only at the cement/tooth interface. A zinc phosphate and an EBA cement showed slits at both interfaces whereas a polycarboxylate cement was the only cement which exhibited good adaption to enamel and dentin, leaving gaps at the cement/alloy interface.

Key-words: Dental materials; fixed partial prostheses

Gudbrand Øilo, Department of Prosthetics, Dental Faculty, University of Oslo, Geitmyrsveien 69, Oslo 4

Previous studies have shown that all types of cements except the EBA cement, exhibit a linear contraction of 0.5–1.0 % when setting under wet conditions (10, 11, 12). Clinical investigations have demonstrated that the thickness of the cement film under cast restorations often exceeds 100 μm (5, 9). A contraction of 0.5–1.0 % in a layer of this thickness may give slits on one or both sides of the cement film which is sufficient in size for bacterial invasion and thus represent a potential infectious hazard. Such slits could also be of importance for the retentive function of the luting layer as the area of cement in interlocking positions could be reduced. However, a contraction in the cement film could be modified by a possible adhesion of the cements to either tooth substance or restorative material.

The purpose of the present investigation was to study the adaption of four different luting cements to enamel, dentin and restorative material in model systems imitating clinical conditions.

Received for publication, December 23, 1977

MATERIALS AND METHODS

Types, trade names, batch numbers and powder liquid ratios of the materials employed are listed in Table 1. The cements were mixed according to the manufacturer's instructions at 23±1 °C.

Pilot study

A replica technique was chosen to study cement adaption between crown and abutment in order to avoid a dehydration of the specimens, i.e. an indirect technique. In a pilot study, a light bodied silicone (Xantopren blue, Bayer, Leverkusen, Germany) was used as the replicating material and an epoxy resin (Durcupan® ACM, Fluka AG, Buchs, Switzerland) as the model material. An inspection of replicas and models showed that slits between cement and the adjacent materials could be seen as projections of the impression material which were reproduced as slits, at the interfaces on the model (Fig. 1). The pilot study also showed that cut and polished surfaces were covered with a layer of grinding debris masking surface details and interfaces (Fig. 2). Etching these surfaces with 1 % solution of phosphoric acid for 10 seconds removed the debris and a superficial layer of the three soluble cements. The cement surface was thus reproduced on the replica as the top level on a rim between tooth and crown (Fig. 3).

The thickness of the cement film varied from less than 50 µm to somewhat more than 200 µm. Slits of constant width were observed along cement films varying more than 100 µm in thickness. The precision of the crowns was therefore not considered in the subsequent study.

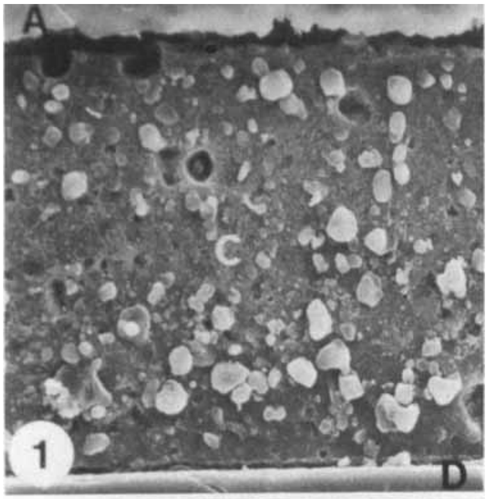
A microscopic inspection of the replicas showed that some of the projections of the impression material corresponding to the slits were curled and rested on the replica (Fig. 9, 10). These projections could probably not be reproduced as slits on the positive model, and therefore an observation of projections was chosen as the method for the present study of cement adaptation.

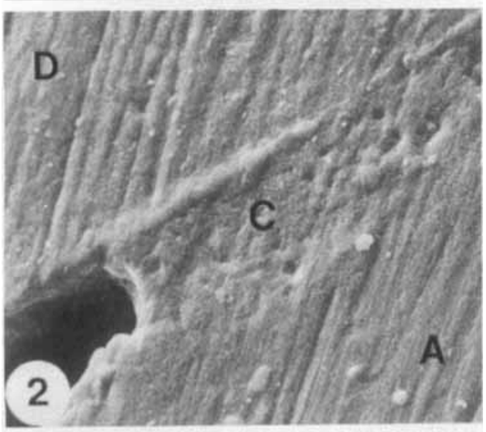
Method I

Twenty extracted human molars and premolars were prepared for complete veneer metal crowns, using diamond instruments

Table 1. *Cements used in the present study*

Type	Name	Manufacturer	Batch no. A	Batch no. B	Powder/liquid-ratio
Zinc phosphate	De Treys Zinc Cement Improved	De Trey Freres S.A. Zürich, Switzerland	Powder NM 40 HK. Liquid Nj 10 BC	Powder SKITBK Liquid SEIIDK	3 g/ml
Polycarboxylate	Durelon	ESPE GmbH Seefeld/Oberbay, Germany.	Powder 593736 Liquid 084162	Powder P7512631 Liquid L7609734	1,5 g/g
Reinforced zink oxide eugenol	Opotow Alumina EBA, Crown & Bridge cement	Teledyne Dental Getz-Opotow Div. Elk Grove Village Illinois, U.S.A.	4801N 6431N	092575	7 g/ml
Composite Resin	Epoxylite® CBA 9080	Lee Pharmaceutical, South El Monte California, U.S.A.	3043BR–1	1074BP–2	2 g/g





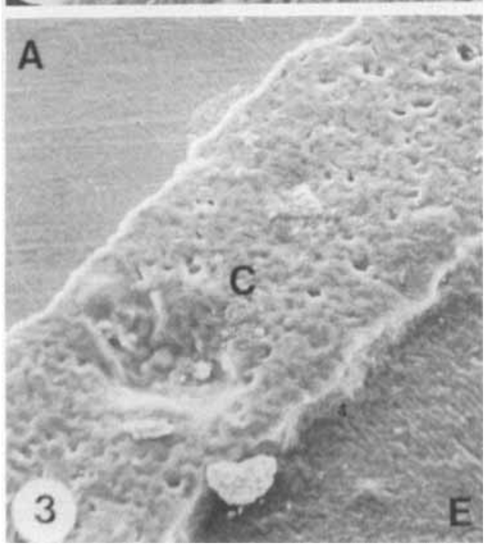


Fig. 1. Positive model of the cement layer (C) between alloy (A) and dentin (D) with slits at both interfaces, X 600.

Fig. 2. Positive model of cement (C), alloy (A) and dentin (D) covered with grinding debris, X 300

Fig. 3. Replica of zinc phosphate cement (C) between alloy (A) and enamel (E), X 200.

---

rotating approximately 20 000 rpm under water spray. Impressions were made with an elastomere in wide copper bands and dies were made of stone. Metal crowns were produced in a silver/palladium alloy (Hvitstøp, K.A. Rasmussen, Hamar, Norway).

The teeth were stored in a 0.2 % aqueous solution of chloramine. Prior to cementation of the crowns they were brushed with a pumice slurry to remove grinding debris (2), rinsed with deionized water and dried with an air-stream at room temperature. The fitting surface of the crowns were sandblasted, filled with cement and placed on teeth with maximum hand pressure. Each of the cements listed in Table 1, batch numbers column A, were used for the cementation of 5 crowns. The cementing procedure including mixing, lasted 3 min. The crowned teeth were stored in 100 % relative humidity for 14 days.

The teeth were then mounted in a silicone mould and embedded in an acrylic resin (Tensol Cement® no. 7, ICI, Plastics Division, Welwyn Garden City, Herts., England). The bloc was prepared using 1 part of component B to 25 parts of component A of the resin. Twenty minutes after start of the mixing the mould was placed in a water bath at 15 °C to reduce the temperature peak during polymerization. After 100 minutes the invested tooth was removed from the water bath, freed from the silicone mould and stored for 24 hours at 100 % relative humidity.

The crown part of the tooth was cut perpendicular to the long axis of the tooth with a diamond wafering blade (*Buhler Ltd.*, Evanston, Il., USA). The cut surfaces of both parts were thereafter ground wet on carborundum paper no. 400 and 600, and lightly polished with an aqueous suspension of aluminium oxide (0.05 μm, Struer Scientific Instruments, Copenhagen, Denmark). The polished specimens were rinsed, blown dry with air, and exposed to 1 % solution of phosphoric acid for 10 seconds to remove grinding debris and crushed cement from the surface of the specimen. The specimens were then rinsed with water and dried with a blast of air at room temperature.

A replica of the polished surface was made using a light bodied silicone material (Xantopren blue, Bayer, Leverkusen, Germany). The replicas were inspected in a stereo microscope (Mak, Reichert, Vienna, Austria) using up to 100 X magnification. Thereafter they were coated with a layer of gold using a Diode-Sputtering System (Polarone Equipment Ltd., Watford, Herts., England), and examined in a scanning electron microscope (SEM) (JEOL J SM – A Scanning Microscope, JEOL Ltd., Tokyo, Japan) using up to 3000 X magnification.

Method II

Twelve extracted human molars were prepared and complete metal crowns were produced as previously described (Method I). The specimens were mounted in a silicone material and a hole was drilled through the crown and the dentin cone, suitable for a brass bolt 1.6 mm in diameter (Fig. 4). The crowns were cemented as previously described and the brass bolt was immediately pressed through the hole and fixed with a nut. The cementing procedure including mixing, lasted 4 minutes. The crowned teeth were stored for 14 days in deionized water at room temperature. Each of the cements listed in Table 1, batch numbers column B, were used for the cementation of 3 crowns.

After 14 days, teeth with crowns were embedded in an epoxy resin (Epofix, Struer Scientific Instruments, Copenhagen, Denmark), and thereafter sectioned longitudinally in two halves. Both surfaces were treated and replicas made as described above (Method I).

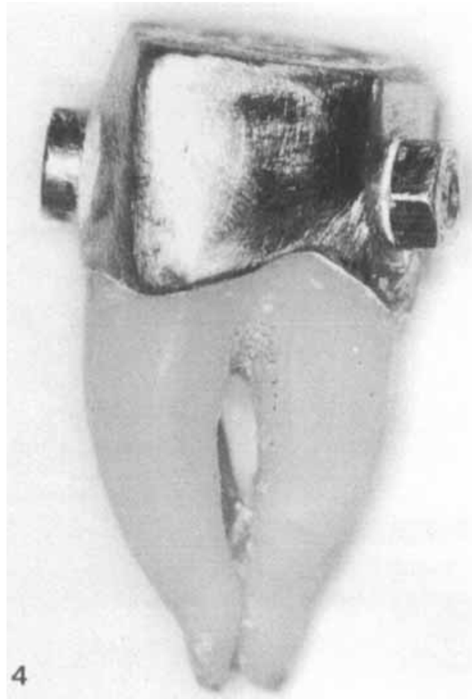


Fig. 4. Tooth with bolted crown.

RESULTS

The inspection of replicas in both microscopes revealed projections of the impression materials at the interfaces between cement and adjacent tooth or restoration material. No difference was seen between the specimens from unbolted (Method I) and bolted (Method II) crowns, and the subsequent description of the projections will therefore not differentiate between the two techniques.

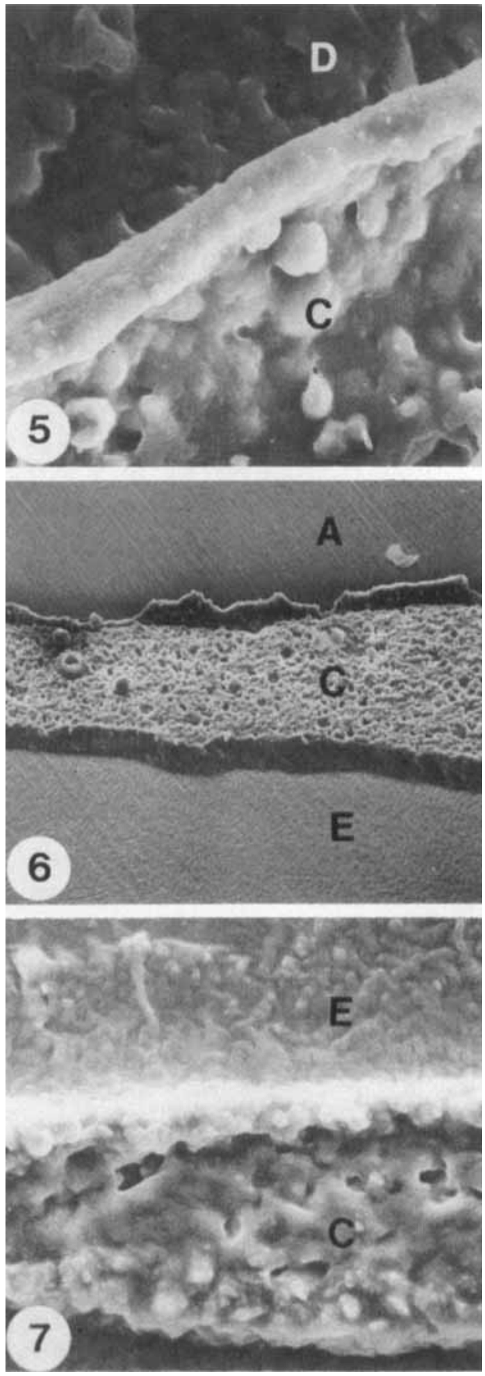


Fig. 5. Projection at the interface between zinc phosphate cement (C) and dentin (D), X 2500.

Fig. 6. Projections showing slits at the interface between polycarboxylate cement (C) and alloy (A). E = enamel. Note the irregular shape of the projections, X 150, 45 ° tilt.

Fig. 7. Projection at the interface between EBA cement (C) and enamel (E), X 500.

Stereo microscopy

Inspection of the replicas showed that the alloy was reproduced as a smooth, glossy surface in distinct contrast to the rough areas representing the cement. In addition, the cement lining usually appeared as an elevated rim between the alloy and the tooth surfaces. The part of the replica representing enamel showed a striped pattern and that of dentin had a "tufty" appearance. On replicas from the specimens with resin cement, no rim was observed but a distinct interface existed between this cement and the adjacent structures.

Feather-edged projections of the impression material at the cement/tooth interface or at the cement/alloy interface could be discerned. On replicas from the zinc phosphate cement and EBA cement such projections occurred frequently along both interfaces, whereas those from the polycarboxylate cement only seemed to have projections at the cement/alloy interfaces. Replicas from the composite resin cement showed projections at the cement/tooth interface only.

The projections varied in height, width and length from scarcely visible tags to long sheets following the interface several millimeters. However, a distinct difference in the extent of projections between the various cements could not be seen.

Scanning electron microscopy

Inspection in the SEM confirmed the observations made with the stereo microscopy. The projections at the interfaces varied considerably in width as well as height and length. Some ended in an evenly, rounded border (Fig. 5), others had an irregular border (Fig. 6) and occasionally sheet-like projections were observed (Fig. 9).

The width of the projections observed in the specimens from zinc phosphate, polycarboxylate and EBA cements varied from less than 1 μm up to 10 μm. In specimens with the composite resin cement projections up to 15 μm wide were observed. No striking relationship appeared to exist between the width of the projections and the thickness of the cement film. On the other hand, the appearance, localization and to a certain degree the extent of the projections, differed between the various cements.

Zinc phosphate cement

Projections were observed most frequently between tooth and cement. They could often be seen as long continuous edges of an irregular, rounded shape (Fig. 5). In some specimens they seemed to cover more than half of the total cement/tooth interface. No difference was observed between the cement/enamel or the cement/dentin interfaces.

Similar projections were observed between the cement and the alloy but they were in general smaller and seemed to cover less of the interfaces.

Polycarboxylate cement

Projections between the cement and the adjacent tooth substance was rare. At the cement/crown interface, however, continuous projections over long distances were commonly observed. They had no typical outline, but varied from irregularly rounded edges to high, tagged or sheet-like projections (Fig. 6).

EBA cement

Projections at the cement/tooth interface were frequently observed, appearing as broad, irregular edges (Fig. 7) intercepted by thin sheets (Fig. 9). Both types occurred at the cement/dentin interface as well as the cement/enamel interface. A few projections were also observed between this cement and the fitting surface of the crown.

Composite resin cement

Projections were observed at the cement/tooth interface only. A thin, regular sheet was often followed continuously in the major part of the specimen (Fig. 8, 10). Again, no difference was seen between projections adjacent to enamel and projections adjacent to dentin.

DISCUSSION

The present study demonstrated that slits occurred frequently at the borders between the cement and the subjacent tooth as well as between the cement and the fitting surface of the crown. Under the present experimental conditions, none of the cements were able to fill the space between crown and abutment completely, but considerable variations between the different cements were noted. Only the polycarboxylate cement showed good adaption to enamel and dentin, leaving gaps only at the alloy side. For the composite resin cement slits were formed only adjacent to tooth substance whereas the zinc phosphate and EBA cements showed slits at both interfaces but with some variations in the extent.

Similar results were obtained with the specimens from method I and II which indicate that the observed slits did not represent artefacts caused by displacement of the crown or dehydration of the specimen. Artefacts from the polishing procedure

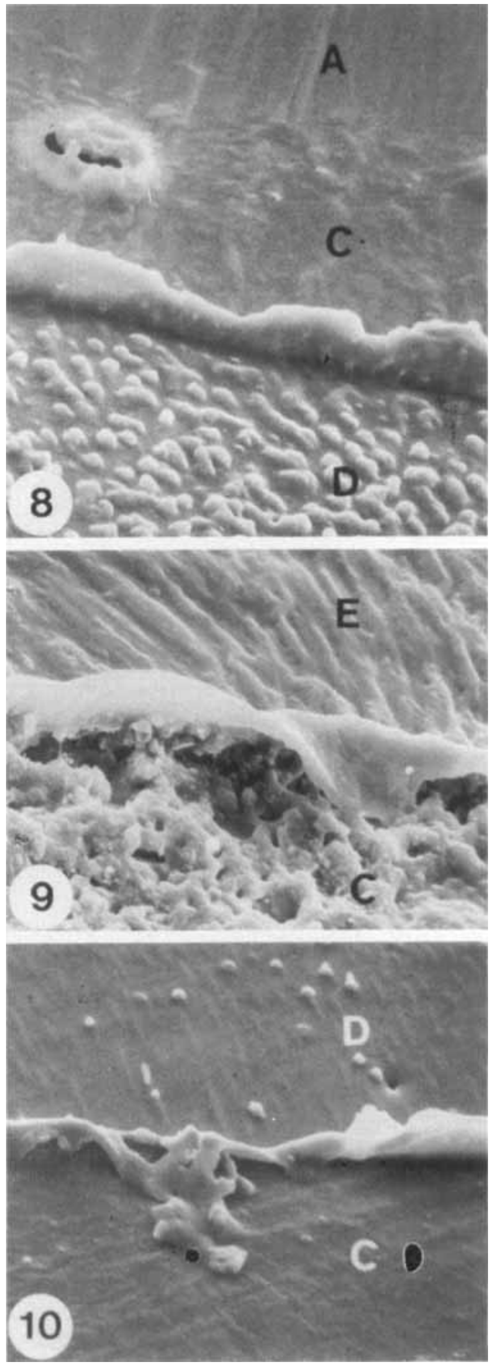


Fig. 8. Projection at the interface between composite resine cement (C) and dentin (D), A = alloy, X 500, 45 ° tilt.

Fig. 9. Deflected projection at the interface between EBA cement (C) and enamel (E), X 1000, 45 ° tilt.

Fig. 10. Distorted projection at the interface between composite resin cement (C) and dentin (D), X 300, 45 ° tilt.

seemed less probable since a superficial layer of the cement was removed during etching. However, the acid etch and variations in the replica technique may have influenced the observed occurrence of slits. The acid may have widened existing slits whereas persisting moisture may have limited the access for the impression material (Fig. 10). The disfiguration (Fig. 9) of some projections may also have limited the recording of existing slits. However, these variations in the method would only be of importance for the extent of slits and not for the observed difference in localization.

The occurrence of slits at the interfaces could depend on an interplay of adhesive forces and tensile stresses (1). None of the materials in the present study created an adhesive bond at both sides of the cement film that could prevent the formation of slits. However, one probable explanation for the differences in slit localization might be the differences in adhesion of these materials to either tooth substance or metal. It has been shown that the composite resin has approximately 10 times larger tensile bond to metal than to bovine dentin (4), and that polycarboxylate cement has a bond of 3–4 MN/m^2 to dentin (7) but only 0.5 MN/m^2 to a silver/palladium alloy (6). The tensile bond of zinc phosphate to dentin has been reported to be very little (7, 8) and negligibile to alloy (6).

The slits could also be a result of air entrapped in the grooves on the tooth surface (3). This could probably be an explanation for some of the smaller slits, but not for the majority of slits which extended over 1 mm or more at the interfaces. In general, the factors which could influence wetting and adhesion such as surface structure, topography, grinding debris or moisture on the adherend and the viscosity of the adhesive, were similar to or even better controlled than in a comparable clinical situation.

As mentioned earlier, the width of the slits seemed to be independent of the cement film thickness. The observed width might, however, indicate that the contraction of the cements in the areas with slits was in the same range as that measured in vitro under dry conditions (10, 11, 12).

REFERENCES

1. Bowen, R.L. Adhesive bonding of various materials to hard tooth tissues. VI: Forces developing in direct-filling materials during hardening. J. Am. Dent. Assoc. 1967, 74, 439–445
2. Dahl, B. Dentine/pulp reactions to full crown preparation procedures. J. Oral Rehab. 1977, 4, 247–254
3. Eick, J.D., Johnson, L.N., Fromer, J.R., Good, R.J. & Neumann, A.W. Surface topography: Its influence on wetting and adhesion in a dental adhesive system. J. Dent. Res. 1972, 51, 780–788
4. Lee, H.L. & Swartz, M.L. Evaluation of a composite resin crown and bridge luting agent. J. Dent. Res. 1972, 51, 756–766
5. McLean, J.W. & von Fraunhofer, J.A. The estimation of cement film thickness by an in vivo technique. Brit. Dent. J. 1971, 131, 107–111
6. Saito, C., Sakai, Y. Node, H. & Fusayama, T. Adhesion of polycarboxylate cements to dental casting alloys. J. Prosthet. Dent. 1976, 35, 543–548
7. Smith, D.C. A review of the zinc polycarboxylate cements. J. Canad. Dent. Assoc. 1971, 1, 22–29
8. Swartz, M.L. & Phillips, R.W. A method of measuring the adhesive characteristics of dental cement. J. Am. Dent. Assoc. 1955, 50, 172–177
9. Windecker, D. & Ohle, K. Einfluss des Herstellungsverfahrens auf den Randschluss von Metallvollkronen. Dtsch. Zahnärztl. Z. 1971, 26, 706–715
10. Øilo, G. Linear dimensional change during setting of a zinc phosphate cement. A methodological study. J. Oral Rehab. 1975, 2, 179–185
11. Øilo, G. Linear dimensional changes during setting of two polycarboxylate cements. J. Oral Rehab. 1976, 3, 161–166
12. Øilo, G. & Ambjørnsen, E. Linear dimensional changes of three luting agents. J. Oral Rehab. 1977, 4, 395–399